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CASEFILE

# OXIDATION RESISTANT CLADDINGS FOR SUPERALLOYS

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### ABSTRACT

The oxidation protection afforded IN-100 and WI-52 superalloys by thin claddings of NiCrAlSi and FeCrAlY alloys was examined primarily at  $1090^{\rm O}$  C. Comparisons were made with commercial aluminide coatings using cyclic furnace and high velocity burner rig tests. In furnace tests, NiCrAlSi on IN-100 and FeCrAlY on WI-52 performed as well or better than two aluminide coatings. Burner rig performance of the FeCrAlY cladding was better than that of the NiCrAlSi cladding on IN-100 and the aluminide coating on WI-52, but less than the aluminide coating on IN-100. An aluminized NiCrAlSi cladding performed better than any coating or cladding.

### OXIDATION RESISTANT CLADDINGS FOR SUPERALLOYS

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### SUMMARY

Claddings of Ni-20Cr-3.5Al-1.2Si (0.051, 0.127, and 0.254 mm thick) and of Fe-25Cr-4.1Al-0.6Y (0.127 and 0.254 mm thick) were diffusion bonded to the nickel-base alloy IN-100 and to the cobalt-base alloy WI-52. The oxidation protection potential of these systems for use in advanced aircraft engines was evaluated by cyclic furnace tests to 400 hours at 1040° and 1090° C (20 hour and one hour cycles) and by Mach 1 burner rig tests at 1040° and 1090° C to 800 hours (1 hour cycles). Similar tests were performed on somewhat thinner aluminide conversion coatings currently used to protect these superalloys in commercial aircraft gas turbine engines. Performance was compared on the basis of visual appearance, weight change, and metallographic analysis.

The NiCrAlSi cladding on IN-100 and the FeCrAlY cladding on WI-52 performed as well or better than the aluminide coatings in furnace tests. In burner rig tests, the claddings generally suffered leading edge losses. The NiCrAlSi cladding on IN-100 performed much worse than did the aluminide coating on IN-100. The FeCrAlY on WI-52 performed a little better than did the aluminide coating on WI-52. At  $1090^{\circ}$  C or below, the claddings, however, never displayed any thermal fatigue cracks as compared to the aluminide coated IN-100 which developed deep cracks in the  $1090^{\circ}$  C tests.

A system involving an aluminized NiCrAlSi cladding on IN-100 exhibited 800 hours protection at 1090° C with much better thermal fatigue resistance than the aluminized coated IN-100. This system performed the best of all the systems evaluated.

#### INTRODUCTION

Aluminide conversion coatings are currently used to protect superalloy components in aircraft gas turbine engines from oxidation, hot corrosion, thermal fatigue, and erosion. The majority of such coatings are applied by diffusion controlled aluminum enrichment of the superalloy surface. Here the substrate chemistry and the processing temperature exert a major influence on coating chemistry, thickness, and properties. Thus, it is difficult to tailor an aluminide coating to resist a particular engine environment.

As engine temperatures increase to improve performance, aluminide conversion coatings offer less potential for providing long time oxidation and thermal fatigue resistance. For this reason, alternate concepts are being examined. One such concept involves overlaying the superalloy surface with an oxidation resistant alloy. Such alloy coatings can be applied by physical vapor deposition, plasma spraying, slurry sintering, or foil cladding (1, 2, 3, 4, 5, 6, 7). Foil cladding requires more preliminary effort and fixturing but it applies a well characterized homogeneous material directly on the superalloy. It thus provides model systems to establish the protection potential and metallurgical interactions for weak, oxidation resistant alloy coatings on strong, less environmentally resistant superalloys.

This paper will review some of the foil cladding studies in which cyclic furnace tests were used to examine oxidation resistance and foil/superalloy interactions. In addition, recent data are presented which reflect the performance of systems tested in high velocity combustion gas environments simulating the temperature transients found in aircraft gas turbine engines. For the sake of comparison, similar data are presented for some of the better commercial aluminide coatings currently used on operational engine components made of the same alloys.

## MATERIALS, SPECIMEN PREPARATION, AND EVALUATION

NiCrAlSi and FeCrAlY foil claddings were applied to IN-100, a typical nickel turbine blade alloy and to WI-52, a typical cobalt turbine vane alloy. The nominal compositions of the claddings were: Ni-20Cr-3.5Al-1.2Si and

Fe-25Cr-4. 1Al-0. 6Y. The nominal compositions of the superalloys were: IN-100; Ni-15Co-9. 5Cr-5. 3Al-4. 3Ti-3. 2Mo, and WI-52; Co-21Cr-11W-2. 2Fe-1. 9Cb-0. 9Si.

For furnace testing, claddings 0.127 mm and 0.254 mm thick of both materials and also 0.051 mm NiCrAlSi were applied to superalloy coupons (50.8 $\times$ 25.4  $\times$ 2.5 mm). Claddings were also applied to 2.5 mm thick coupons of the cladding material (i.e., cladding foil on cladding sheet coupons) to obtain base line furnace oxidation data. Furnace tests were conducted at  $1040^{\circ}$  and  $1090^{\circ}$  C ( $\pm 5^{\circ}$  C control) using twenty and in some cases one hour exposure cycles. For high velocity burner rig testing, 0.127 mm claddings only were applied to the major surfaces of  $101.6 \times 25.4 \times 6.3$  mm bars having one long edge (the leading edge in test) tapered with a  $45^{\circ}$  included angle and a 0.075 mm leading edge radius (8). Mach 1 burner rig tests were conducted at  $1040^{\circ}$  and  $1090^{\circ}$  C ( $\pm 8^{\circ}$  C control) using 1 hour exposures followed by air blast quenching to below  $100^{\circ}$  C in 3 minutes.

The procedures used in applying the alloy claddings included specimen preparation, assembly, hot isostatic gas pressure bonding, and final finishing (5,6). The parameters used in bonding were 2 hours at  $1090^{\circ}$  C and 103-138 MN/m $^2$  (15 000-20 000 psi) helium pressure. The assembly layout for the more complex burner rig specimen is shown in Fig. 1. After bonding and removal from the fixture, the heavy cladding alloy back up plate was machined to 0.03 mm thickness. The commercial aluminide coatings were vendor applied but being proprietary, no information as to processing conditions was available.

System performance was evaluated primarily on the basis of weight change, visual appearance, and metallographic change. Compositional changes were selectively determined by electron microprobe analysis, and the surface scales formed were determined by combined X-ray diffraction and flourescence analyses.

### RESULTS AND DISCUSSION

Furnace exposure results for NiCrAlSi and FeCrAlY claddings on IN-100 and WI-52 will be presented and the better systems compared to aluminide coatings. Burner rig performance of both claddings and coatings will also be compared. Recent promising results on an aluminized NiCrAlSi cladding system for IN-100 also will be presented.

#### A. Furnace Test Results

## NiCrAlSi Clad IN-100 and WI-52

Weight change results of furnace tests on NiCrAlSi clad IN-100 and WI-52 at 1090°C (20 hour exposure cycles) are shown in Fig. 2. (Remember that weight gains are due to oxygen pick up; subsequent or concurrent spalling of the metal oxide with or without vaporization produces weight losses. Since these mechanisms can be active at the same time, many systems show an initial gain and then a turnaround as spall weight and/or vaporization exceeds oxygen pick up. Thus, weight change data represent only a qualitative index of oxidation behavior and must be supplemented by other data such as metallography.) The results in Fig. 2 show that the clad-cladding alloy was quite oxidation resistant in that it gained weight in forming a protective oxide and then little further weight change occurred. While the NiCrAlSi clad on IN-100 did show a slight turnaround primarily due to spalling, it was more protective than on WI-52. The bare IN-100 lost weight rapidly but bare WI-52 (data not shown) lost weight even more rapidly.

Cladding thickness exerted little influence on the weight change behavior of the NiCrAlSi clad IN-100 at 1090°C. However, only the thickest cladding (0.254 mm) on WI-52 did not show a total weight loss even though spalling caused a turnaround in weight change after about 120 hours.

Exposure at 1040° C resulted in more protective behavior for both cladding systems for times to 400 hours. At this temperature, the influence of cycle frequency was examined on the more oxidation resistant system, NiCrAlSi clad IN-100. Increasing exposure cycles from 20 hour to 1 hour exposures increased the rate of degradation considerably in a fashion similar to that observed on aluminide coated B-1900 nickel alloy (unpublished data of S. R. Levine, NASA; Lewis Research Center). For example, for the 0.127 mm cladding on IN-100, the weight change turnaround occurred after 20 hours with 1 hour cycles, but only after 200 hours with 20 hour cycles. This sensitivity to cycle frequency can be expected to be more severe at higher temperatures.

Metallographic cross sections of the NiCrAlSi cladding on IN-100 in Fig. 3 show that this system is relatively unaffected by 200 hour cyclic furnace oxidation at  $1090^{\circ}$  C, especially for claddings of 0.127 mm and thicker. Even the 0.051 mm cladding is still in good condition but here and in the case of the 0.127 mm cladding, complete interdiffusional penetration of the cladding by cobalt, titanium, and molybdenum occurred during the 200 hours of testing. The minor difference in the oxidation performance between clad IN-100 and the clad-cladding alloy is attributed to such interdiffusion. This interdiffusion was detected by electron microprobe analyses but is not readily apparent metallographically, however, aside from the destabilization of the gamma/gamma prime microstructure of the IN-100.

NiCrAlSi clad WI-52 (fig. 3) shows considerable surface oxide penetration and internal oxidation in the 0.127 mm thick cladding after only 120 hours of test. A faint front of interdiffused substrate elements is indicated by the carbide free region just below the cladding in WI-52. Aluminum was also observed to diffuse into the substrate. In this system, only the 0.254 mm cladding appears to have resisted oxidation as well as the 0.127 mm NiCrAlSi clad IN-100. It is believed that this is primarily because the surface of the thick, 0.254 mm, cladding was not as affected by loss of aluminum or ingress of substrate elements within the test time. These changes are believed to be the cause of the poorer oxidation resistance of NiCrAlSi cladding on WI-52 than on IN-100.

## FeCrAlY Clad IN-100 and WI-52

Only 0. 127 amd 0. 254 mm thick FeCrAlY claddings were evaluated on IN-100 and WI-52. As shown by the weight change data in Fig. 4, at 1090° C (20 hour cycles) the weight change behavior of the 0. 127 mm FeCrAlY clad as well as that 0. 254 mm clad WI-52 was almost identical to that of the FeCrAlY clad FeCrAlY. The clad IN-100, however, showed more rapid weight gains accompanied by significant spalling. The bare IN-100 and WI-52 lost weight rapidly as previously mentioned. The thicker (0. 254 mm) cladding on IN-100 and WI-52 performed similarly to the clad-cladding alloy

at 1090° C. Again, the lower exposure temperature of 1040° C resulted in less oxidation attack for the claddings on both substrates. The FeCrAlY cladding on WI-52, furthermore, showed only minor differences in weight change at 1090° C whether 1 or 20 hour cycles were used and only after 340 hours did the specimen given 1 hour cycles start to show a rapid weight loss.

Figure 5 shows the cross-sectional microstructure of the 0.127 mm claddings on both IN-100 and WI-52 after 200 hours (20 hour cycles) at 1090°C. The extensive interdiffusion between the body centered cubic FeCrAlY cladding and the IN-100 substrate resulted in destabilization of the gamma/gamma prime microstructure of the substrate and sufficient nickel penetration to stabilize the high temperature face centered cubic structure and to produce twins. Cobalt, titanium (especially at 1090°C), and molbydenum were major diffusing elements entering the cladding. Such interdiffusion is believed to be responsible for the higher weight gains caused by grain boundary and internal oxidation as compared to the clad-cladding alloy. Localized deep oxide penetration was observed to extend into the cladding and the extent of penetration increased with both time and temperature so that in some cases the oxide completely penetrated the cladding. Time, however, had only a minor influence on the depth of the destabilized zone in the IN-100 substrate.

Metallographically, the interdiffusion in the FeCrAlY clad WI-52 specimen appears slight in Fig. 5. However, the white globular particles (primarily chromium carbide containing tungsten, iron, and cobalt) were seen at the cladding/WI-52 interface as well as the cladding surface suggesting that interdiffusion was rather extensive. After oxidation at both temperatures, the extent and degree of cobalt diffusion into the FeCrAlY on WI-52 was about as great as that of nickel diffusion into the clad IN-100.

As in the FeCrAlY clad IN-100, deep local oxide penetration of the FeCrAlY clad WI-52 was observed after  $1090^{\rm O}$  C oxidation. These systems appear to be very oxidation resistant at  $1040^{\rm O}$  C, but such localized attack zones indicate that life may not be much greater than 400 hours at  $1090^{\rm O}$  C. While specific elements have not been connected with the decrease in oxidation resistance of the FeCrAlY clad systems, dilution produced by outward

substrate element diffusion, inward diffusion of cladding elements and/or cycle induced spalling of the protective alpha aluminum oxide occur to the extent that less protective  $\operatorname{NiCr}_2O_4$  and  $\operatorname{CoCr}_2O_4$  spinels form.

# Claddings Compared to Aluminide Coatings

In Fig. 6 metallographic and furnace weight change data obtained after  $1090^{\circ}$  C furnace tests on the commercial aluminide coatings are compared with similar data for the most protective claddings on each substrate—NiCrAlSi on IN-100 and FeCrAlY on WI-52. The comparisons in Fig. 6 indicate that both the microstructure and weight changes of that coating and cladding on IN-100 are very similar after 200 hours, 20 hour cycles, at  $1090^{\circ}$  C. Here both protection systems are approximately the same thickness. The FeCrAlY cladding on WI-52 is in much better condition that the failed aluminide coating but it was about twice as thick in the deposited condition. The ease in controlling thickness is a real benefit of the cladding approach.

### B. BURNER RIG TEST RESULTS

# Individual Claddings and Coatings

The most promising cladding systems based on furnace testing were the NiCrAlSi clad IN-100 and the FeCrAlY clad WI-52. FeCrAlY clad IN-100 also appeared to have some potential. These systems were subjected to Mach 1 burner rig testing at both  $1040^{\circ}$  and  $1090^{\circ}$  C using one hour exposure cycles followed by air blast quenching. Such testing imposed significantly greater thermal stress on the protection system and the surface oxides, especially at the leading edges of the burner rig specimens. It also introduced into the evaluation a high velocity combustion gas environment similar to that found in aircraft gas turbine engines. For the sake of comparison, commercial aluminide coated IN-100 and WI-52 (the same coatings as furnace tested) were also subjected to these tests.

On the basis of weight change in high velocity burner oxidation, Fig. 7(A), the aluminide coated IN-100 performed much better at both temperatures than the thicker NiCrAlSi cladding. Surprisingly, the FeCrAlY cladding on IN-100

which was only tested at 1040°C because of the high weight gains shown in furnace oxidation at 1090°C, resisted high velocity oxidation much better than expected although not quite as good as the aluminide coating. The NiCrAlSi cladding lost weight very rapidly in these tests and performed less satisfactorily than even the unprotected IN-100 on a weight change basis. In the case of WI-52, Fig. 7(B), the bare alloy is extremely poor in high velocity oxidation resistance. Here, at both temperatures, the thicker FeCrAlY cladding showed longer times to weight loss than did the aluminide coating.

Visual and metallographic examination of the maximum attack zones on the clad burner rig specimens showed that the large weight losses were generally due to the loss of cladding from the leading edges. While such losses of cladding were observed even at  $1040^{\circ}$  C, in all cases the sides of the test bars were generally still covered with useful cladding material. In comparison, the aluminide coated IN-100 was completely intact after 240 hours of testing at  $1040^{\circ}$  C—the total time of that test. Aluminide coated WI-52 showed some weight losses but visual failure was observed only after 300 hours of testing at  $1040^{\circ}$  C.

Metallographic examiniation and visual observation of the clads tested at  $1090^{\circ}$  C showed that the leading edge attack was more severe and some general attack could also be seen. No thermal fatigue cracks were observed at this temperature (or at  $1040^{\circ}$  C) for the test times employed. In contrast, the aluminide coating developed thermal fatigue cracks between 40 and 140 hours of test. Metallographic examination of aluminide coated IN-100 after 300 hours of test indicated that the observed weight losses were mainly due to localized oxidation and spalling at the cracks which by this time extended deep into the IN-100 substrate. In the noncracked areas, the aluminide coating still appeared sound. Aluminized WI-52 failed rapidly by general oxidating attack at  $1090^{\circ}$  C.

The decrease in high velocity protection afforded by the claddings as compared to their behavior in furnace tests is primarily attributed to their relatively low aluminum content. Aluminum is the prime element needed to form the protective alpha aluminum oxide scale. The 20 hour cyclic and

even the one hour cyclic furnace tests do not stress the oxide scales as much as the rapid heating-cooling of the burner rig tests. Thus, in burner rig testing, aluminum loss is accelerated and a point is reached more rapidly where sufficient aluminum is no longer present to form alumina and only the less protective spinel type oxides can form. These spinel oxides spall more readily and weight losses increase.

In no case were thermal fatigue cracks observed in the tested clad systems. This indicated that while the high velocity oxidation and/or erosion resistance of the clads was in some cases about equal and in others less than the coatings, their thermal fatigue resistance was markedly superior.

### Aluminized NiCrAlSi Claddings

Since the soft, ductile NiCrAlSi and FeCrAlY claddings showed superior resistance to thermal fatigue cracking and since, the harder, more brittle aluminide coatings on IN-100 resisted oxidation erosion better but cracked and permitted cracks to propagate into the substrate, some NiCrAlSi clad IN-100 burner specimens were aluminized to determine if the benefits of both protection systems could be combined.

As shown in Fig. 8, aluminizing of the NiCrAlSi cladding resulted in a markedly improved protection system for IN-100. Over 800 hours of protection was afforded IN-100 in 1090° C burner rig tests. Based on the time to show a weight change turnaround, the aluminized clad (700 hours) was four to five times as protective as the commercial aluminide coating (160 hours).

Figure 9(a) shows the external appearance and Fig. 9(b) shows selected cross-sectional microstructures of the aluminized NiCrAlSi clad IN-100 burner specimen after 800 hours of Mach I testing at  $1090^{\circ}$  C. While some cracks were visually ovserved at the trailing edge (there is about a  $30^{\circ}$  C increase in temperature from the leading to the trailing edge in these tests), no other evidence of serious degradation is evident. The photomicrographs show that oxide penetration has occurred into the cladding layer at the leading edge but no substrate attack was observed. All of the NiAl layer in this high thermal stress region has been converted by loss of aluminum to

gamma prime (Ni<sub>3</sub>Al) and gamma (nickel solid solution). On the side of the bar in a lower thermal stress region where spalling of the alpha alumina scale was less, the NiAl layer is intact but shows striations—indicative that some aluminum loss has taken place to produce this nickel-rich martensitic structure (10). The cladding is also intact here. The trailing edge is the hottest part of the burner rig specimens (about 1113°C). This edge contains the cladding backup plate. Some cracks were visually observed in this part of the specimen.

The benefit of the aluminized NiCrAlSi cladding on IN-100's thermal fatigue resistance in the burner rig tests can be clearly seen in Fig. 10. The unprotected IN-100 showed early thermal fatigue cracking (within 40 hours) during  $1090^{\circ}$  C exposure using 1 hour cycles followed by air blast quenching. While the aluminide coating extended the time to the first visible crack by a factor of about two (40 to 140 hours), the aluminized NiCrAlSi cladding (280-420 hours) extended the time to first crack by a factor of about nine.

These promising findings are quite recent. The full benefits of the cladding plus aluminizing approach, its drawbacks, and its extrapolation to other alloy coatings have yet to be explored. Also, interdiffusional, compositional, microstructural, and oxide scale characterization are needed to fully explain the reasons for the improved performance.

### SUMMARY OF RESULTS AND CONCLUDING REMARKS

The NiCrAlSi cladding on IN-100 and the FeCrAlY cladding on WI-52 exhibited good oxidation resistance in cyclic furnace tests at temperatures to  $1090^{\circ}$  C. In such tests, especially the thicker claddings (0. 127 and 0. 254 mm) performed as well or better than somewhat thinner commercial aluminide coatings. Where examined, cycle frequency increases, from 20 to 1 hour exposures, shortened the time to weight change turnaround (i.e., when the spall metal oxide weight loss exceeded oxygen weight gain). This same behavior has been observed on aluminide coatings tested at these temperatures.

In high velocity, Mach I burner rig tests using rapid cooling after each one hour exposure cycle, the claddings were less protective. Weight losses were generally due to the loss of cladding from the small radius, highly thermal stressed leading edges of the test specimens. The FeCrAlY cladding performed better on both IN-100 and on WI-52 than did the NiCrAlSi cladding. The NiCrAlSi cladding performance on IN-100 was significantly inferior to the commercial aluminide coating while the FeCrAlY cladding on WI-52 was somewhat better than a thinner, commercial aluminide coating. The thermal fatigue resistance of the claddings was very good, however. In all of these tests, no cracks were observed in the claddings within the test times.

In both furnace and burner rig tests, interdiffusion of cladding and substrate elements is believed to cause a decrease in system oxidation resistance. The determination of the most detrimental elements to cladding performance, however, will require additional study.

By aluminizing the NiCrAlSi cladding on IN-100, a system was achieved that withstood at least 800 hours of Mach I burner rig testing at 1090° C. This sytem exceeded the oxidation and thermal fatigue resistance of one of the best commercial aluminide coatings by over a factor of three or more.

The primary cause for improvement in thermal fatigue resistance is believed to be the existence of a rather ductile oxidation resistant layer of aluminum enriched cladding under the external aluminide coating. In conventional aluminide coatings on superalloys, a hard, carbide rich zone is typically found here. Benefits may also be derived from the conversion of the relatively simple NiCrAlSi alloy to an aluminide. This aluminide would be expected to contain little of the strengthening elements found in IN-100. Indications are that many such elements degrade the oxidation resistance of aluminide coatings.

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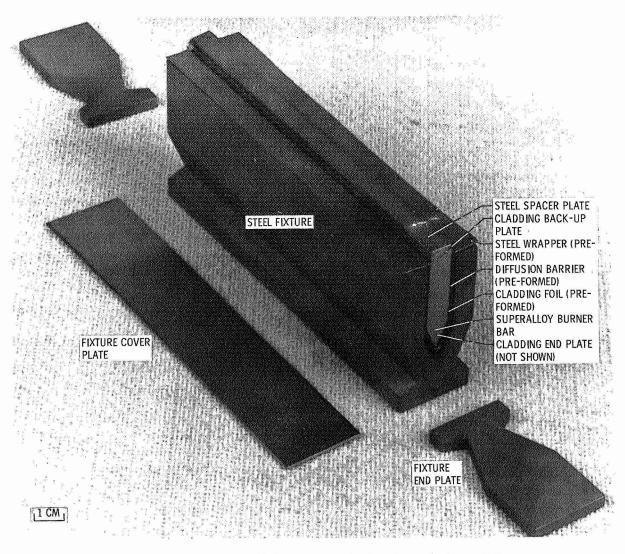


Figure 1. - Component assembly for gas pressure bonding of superalloy burner bars.

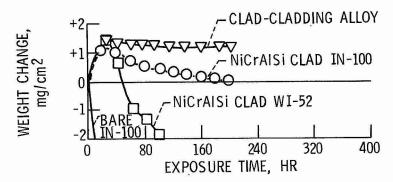


Figure 2. - Weight change of 0.127 mm Ni - 20 Cr - 3.5 Al - 1.2 Si clad substrates during 20 hour cyclic furnace exposure testing at 1090 C.

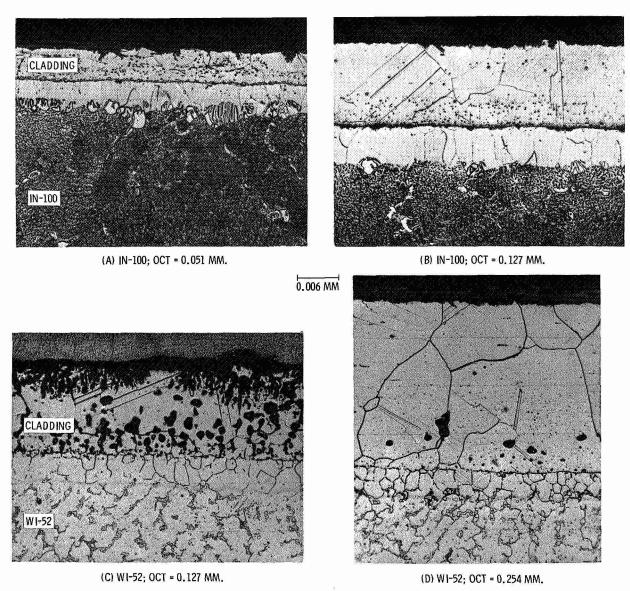


Figure 3. - Microstructures of Ni-20Cr-3.5Al-1.2Si clad IN-100 and WI-52 after ten, 20-hour cyclic furnace oxidation exposures at 1090° C. The 0.127 mm clad WI-52 specimen was tested for six, 20-hour exposures. (OCT = Original cladding thickness.)

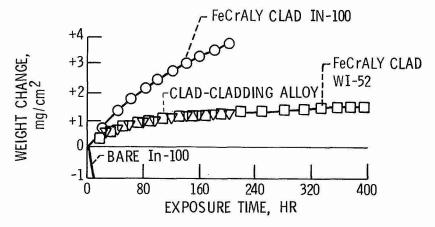


Figure 4. - Weight change of 0.127 mm Fe-25 Cr-4.1 Al-0.6 Y clad substrates during furnace oxidation at  $1090^{\circ}$  C (20 hr cycles).

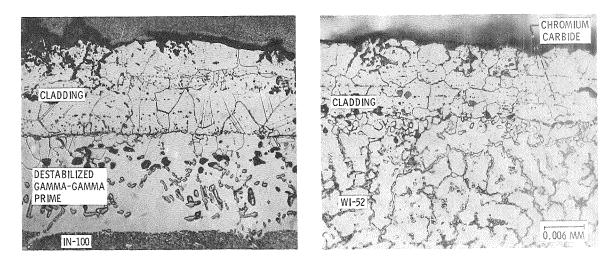


Figure 5. - Microstructures of 0.127 mm Fe-25Cr-4.1AI-0.6Y clad IN-100 and WI-52 after ten, 20-hour cyclic furnace oxidation exposures at 1090° C. X250.

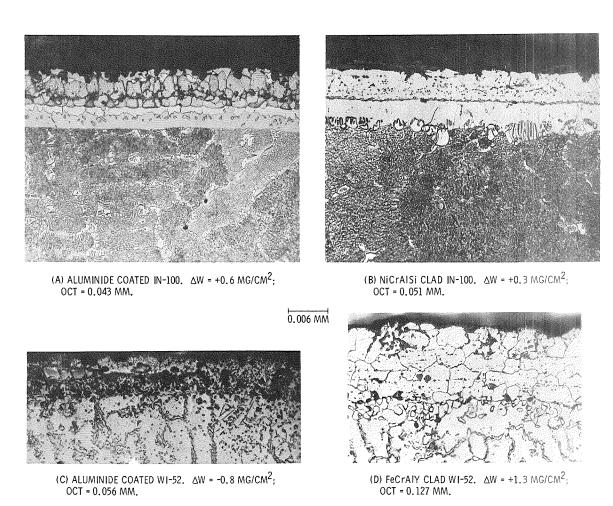


Figure 6. - Microstructures of coated and clad superalloys after 200 hours (20 hour cycles) furnace oxidation at  $1090^{\circ}$  C. X250. (OCT = Original coating or cladding thickness.)

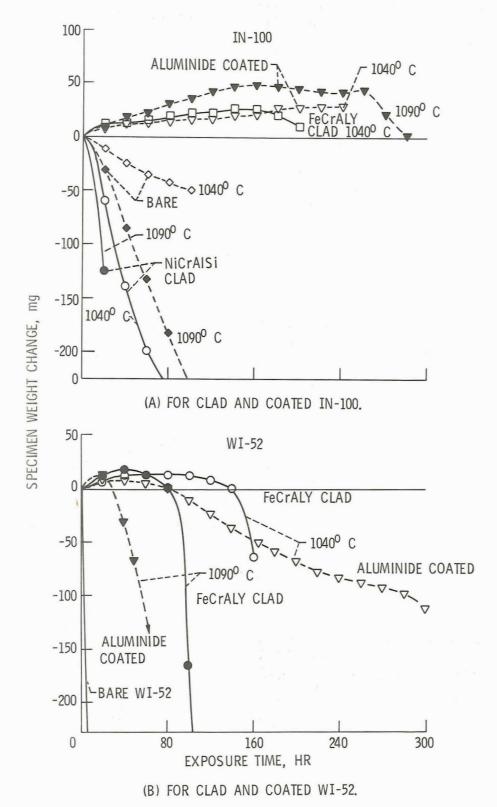


Figure 7. - Comparison of 1 hour, Mach 1 cyclic burner rig test weight changes.

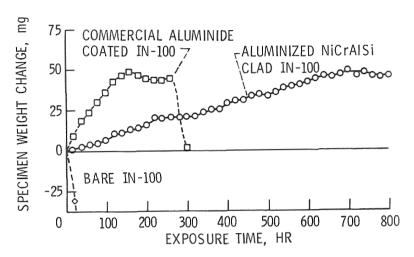
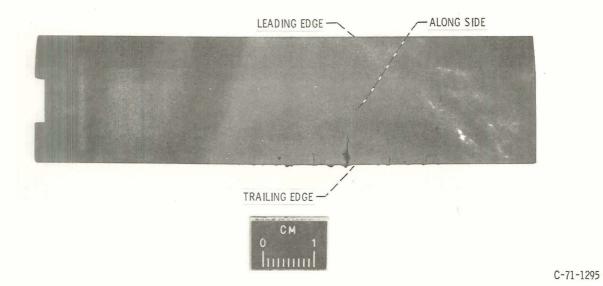
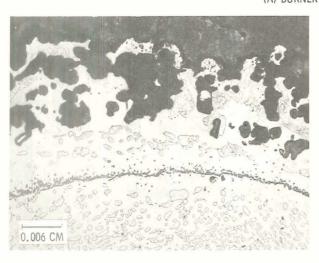
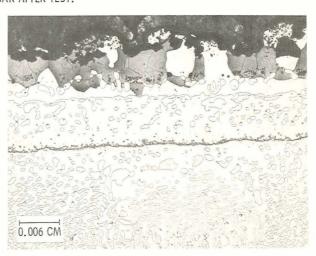


Figure 8. - Comparison of Mach 1, 1-hour cyclic burner 1090<sup>0</sup> C test weight changes for aluminide coated and aluminized clad IN-100.



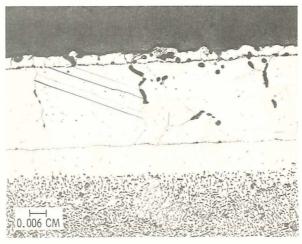
### (A) BURNER BAR AFTER TEST.





LEADING EDGE,  $T_{max} \approx 1085^{\circ}$  C.

ALONG SIDE,  $T_{\mbox{max}} \approx 1099^{\circ}$  C.



TRAILING EDGE,  $T_{max} \approx 1113^{\circ}$  C.

(B) MICROSTRUCTURES WITHIN MAXIMUM ATTACK ZONE.

Figure 9. - Aluminized Ni-20Cr-3.5Al-1.2 Si clad In-100 burner bar after cycle burner testing at  $1090^{\circ}$  C and Mach 1 for 800 hours.

### MACH 1 BURNER RIG TEST AT 1090° C - 1 HOUR CYCLES

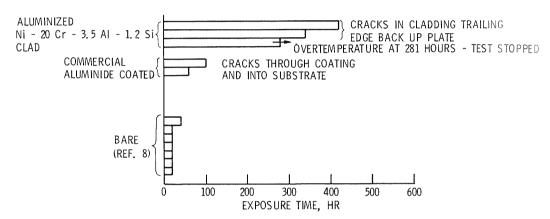


Figure 10. - Time to first observable thermal fatigue cracking of bare and protected IN-100. (Each bar represents one specimen.)